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**The Ability of AMSTAR Dechlorination Solution to Remove and Degrade PCBs  
From Contaminated Surfaces**

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**Prepared for ESTCP**

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## Introduction

Polychlorinated biphenyls (PCBs) are a group of synthetic aromatic compounds with the general formula  $C_{12}H_{10-x}Cl_x$  that were historically used in industrial paints, caulking material and adhesives, as their properties enhanced structural integrity, reduced flammability and boosted antifungal properties. Although the United States Environmental Protection Agency (USEPA) has banned the manufacture of PCBs since 1979, they have been found in at least 500 of the 1,598 National Priorities List (Superfund) sites identified by the USEPA. Prior to the USEPA's ban on PCB production, PCBs were commonly used as additives in paints and asphalt-based adhesives that were subsequently applied to a variety of structures. Government facilities constructed as early as 1930 utilized PCB-containing binders or PCB-containing paints, which are now leaching into the environment and posing ecological and worker health concerns.

In 2006, a commercially available product known as AMSTAR Dechlorination Solution was tested at NASA's Kennedy Space Center for its ability to remove and degrade PCBs from structural materials. This evaluation was requested by the Environmental Security Technology Certification Program (ESTCP) as an add-on to ESTCP funded research (Project 06 E-CP4-025) evaluating the ability of NASA's Bimetallic Treatment System (BTS) to remove and degrade PCBs from structural materials. The results of the laboratory testing are to be used to determine if a side-by-side field-scale test comparing BTS to AMSTAR was warranted.

A recommended sampling and analysis testing program was submitted to ESTCP that included triplicate screening of AMSTAR's PCB dechlorination capabilities on a variety of surfaces including glass, bare metal, and painted metal coupons. The test procedures, analytical techniques and results obtained are presented in this interim report to ESTCP.

## Experimental

AMSTAR Dechlorination Solution was tested in a number of experiments to determine its ability to remove and/or dechlorinate PCBs from different environments: glass vials, bare metal strips, and painted metal coupons. All experiments were performed using PCB mixture Aroclor 1254 which has a variety of PCB congeners. All samples were analyzed using a gas chromatograph coupled with a mass spectrometer (GC-MS).

### Glass Vials

Initially, a series of tests was performed in glass vials to evaluate the degradation of the PCB "finger print" that could be expected with the AMSTAR product. AMSTAR Dechlorination Solution is a patented, water-based material that has a composition

consisting of sodium silicate, polyethylene glycol, and a nonionic surfactant. The product's vendor advocates that AMSTAR extracts PCBs from solid surfaces and destroys them by converting the PCBs to products that are thought to be non-hazardous. The glass vial experiments were carried out to look for degradation end-products using a GC-MS. The product's ability to "extract" PCBs from the vial was not the focus of the glass vial study. This vial experiment was only looking for the product's degradation capabilities.

A 5000 mg/L in methanol standard of PCB mixture Aroclor 1254 was diluted in hexane to make a stock standard solution of 100 mg/L for use in the glass vial experimental setup. Five mL of the 100 mg/L standard were pipetted into a set of glass vials and allowed to dry so PCBs were deposited on the bottom of the glass vial. To these vials, 10 mL of AMSTAR Dechlorination Solution were added and allowed to sit, capped, to test the degradation properties of the AMSTAR product over an extended period of time. Glass vials were sacrificed for analysis ( See Figure 1) after being allowed to react for periods of one day, two days, four days, or nine days. At the end of the designated time period, each sample was extracted from a glass vial with five mL of hexane, shaken for two minutes and allowed to separate. The hexane layer that separated on top appeared cloudy and gelatinous, presumably an emulsion of hexane, water and surfactant. The cloudy hexane layer was pipetted off the top and treated with two grams of anhydrous sodium sulfate powder to clean and remove any water or surfactant from the extract, thus allowing for clean hexane separation.

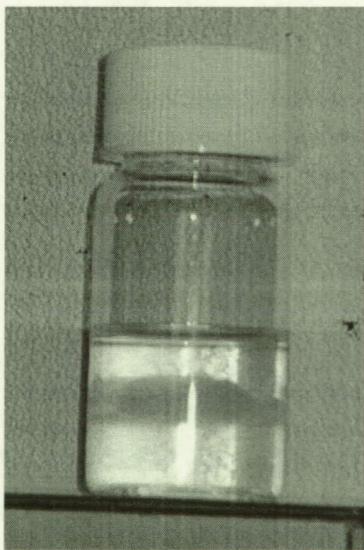


Figure 1: Glass Vial Study with Hexane Layer on Top During Extraction

Control samples were created and handled using the same procedure as described above except no AMSTAR was applied to the control vials. Laboratory blanks were routinely run every time a set of samples were analyzed on the GC-MS.



## Metal Strips

A second series of experiments was set up to determine the ability of AMSTAR to extract PCBs from a bare metal surface. These metal strip experiments also allowed for further testing of AMSTAR's dechlorination capability. The AMSTAR product dries as a white residue that is difficult to scrape completely from a metal coupon. For the metal strip experiments, the AMSTAR product, once dried post-application, was rinsed in a water bath to allow the residue to re-solubilize in water. The assumption was that any reaction involving Amstar producing innocuous end-products will have occurred post-AMSTAR application and simply re-dissolving or reconstituting the residue back into water should not reformulate hydrophobic PCBs. Any un-reacted PCBs, therefore, would remain on the metal surface as opposed to entering into the water continuum.

Strips of steel were cut with dimensions 1.25 in. x 0.5 in. from 1/16 in. thick stock sheet metal. Each of these strips was dosed with 100  $\mu$ L of 5000 mg/L in methanol PCB standard and allowed to dry. (See Figure 2) Each strip was then treated with AMSTAR product by one of two methods. In the first method, the strips were coated with 1 mL of AMSTAR and allowed to air dry for 3 days. These strips were then soaked in a vial full of water to reconstitute the AMSTAR residue before being soaked in hexane to extract the remaining PCBs on the metal strips. The second method took the PCB-coated metal strips and placed them in a capped vial of 10 mL of AMSTAR to keep the product in liquid form. These strips were then rinsed with water to remove any remaining liquid AMSTAR before being soaked in 10 mL of hexane to extract any PCBs remaining on the metal.

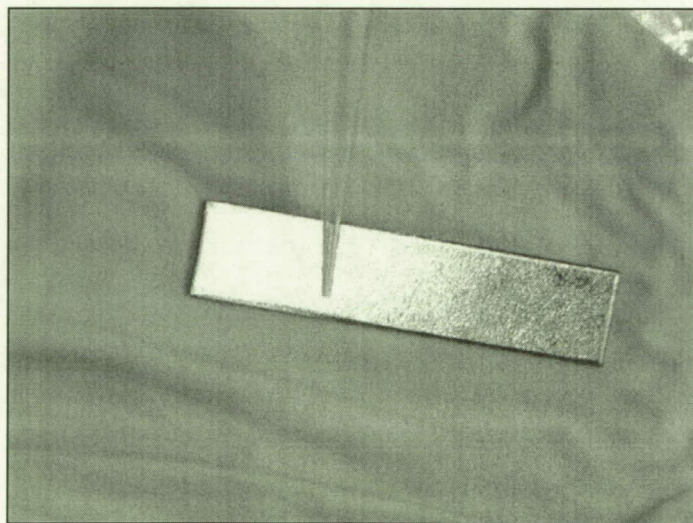


Figure 2: Application of PCBs to Metal Strips

Control samples were carried through the entire procedure as described above except no AMSTAR was applied to the control strips. Blanks were routinely run every time a set of samples were analyzed on the GC-MS.

In an attempt to recover any PCBs removed from the metal strips and to check for possible dechlorination, the reconstituted AMSTAR solution was concentrated and extracted into hexane. As with the glass vial experiments, the hexane was cleaned with anhydrous sodium sulfate if the extract looked cloudy. Again, control samples were taken through the entire extraction procedure.

### Painted Metal Coupons

Originally, painted, PCB-containing metal plates remaining from NASA's Apollo program were to be tested using the AMSTAR product. However, because of significant disparities in PCB concentrations across the plates (variations from 500-5000 mg/Kg across a three-foot square panel), a more controlled laboratory procedure was selected. Metal coupons were painted with a paint primer and a known amount of PCBs were added to the drying paint. This testing procedure differed from the bare metal application in that the test aimed to evaluate AMSTAR's ability to extract PCBs from a more complex matrix. AMSTAR was given an advantage in this test setup because the PCBs were not mixed with the paint on the coupons. Instead, they were placed on the top surface of the paint as it was drying, and not mixed within the paint, giving the AMSTAR product the greatest advantage for extraction. This test was again designed to look at AMSTAR's extraction capabilities and not degradation abilities.

Steel coupons were cut with dimensions 2 in. x 2 in. from 1/16 in. thick stock sheet metal. Each coupon was painted with white primer (See Figure 3). The paint was allowed to dry until a film formed and was then dosed with 100  $\mu$ L of 5000 mg/L PCBs. Once the painted surface of the coupon was dry, it was coated with three mL of AMSTAR product and allowed to dry for three days. After the AMSTAR dried, the coupon was soaked in water to reconstitute the AMSTAR residue and remove it from the painted surface. The paint was then scraped off the coupon and sonicated in 10 mL of either hexane or ethanol for extraction of remaining PCBs. Samples extracted in ethanol were later transferred to hexane prior to injection into the GC-MS in an effort to cleanup the sample. Previous work with paint extracts has shown that the transfer from ethanol to hexane removes a significant amount of analytical interferants derived from the paint (BTS Application at Marshall Space Flight Center Final Report, 2006). With this procedure, the sample is first extracted and sonicated in ethanol before a small amount of water is added, followed by the addition of the final solvent, hexane.

Control samples were carried through the entire procedure (including water soak and hexane extraction) as described above except no AMSTAR was applied to the control paint strips. Blanks were routinely run every time a set of samples were analyzed on the GC-MS.



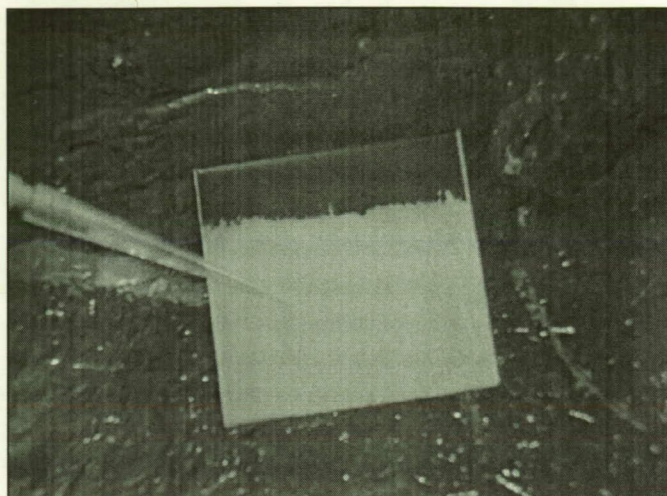


Figure 3: Painted Metal Coupon with PCBs  
Applied to Surface

## Results

Chromatograms and other supporting data are presented in the Appendix. All results are presented in concentration units of milligrams per liter (mg/L), with the mass of PCBs extracted into a known volume of hexane prior to GC-MS analysis. Complete PCB recovery would yield 100 mg/L for the glass vial study and 50 mg/L for both the bare metal strip tests and the painted metal coupon tests. The control values should be used as the bench-mark for the most recoverable amount of PCBs given the extraction procedure used.

### Glass Vials

Studies with precipitated PCBs in a glass vial and liquid AMSTAR show no discernable trend in the reduction of PCB concentration over time, as shown in Table 1. All sampling times show an average recovery of greater than 90% of the initial PCBs with respect to the control samples. The GC-MS spectra of the treated samples are nearly identical to those of the non-treated control and do not show any signs of degradation byproduct formation.

**Table 1: PCBs in glass vials**

	Sample 1	Sample 2	Sample 3	Average	Std Dev
Control	88.01	114.97	86.02	96.33	16.17
1 day	91.23	80.30	87.35	86.29	5.54
2 day	98.73	89.69	89.29	92.57	5.34
4 day	95.51	101.49	94.14	97.05	3.91
9 day	*	97.34	111.22	104.28	9.81
*Sample compromised					
**All concentrations in mg/L.					

## Metal Strips

The data supporting the ability of AMSTAR to extract PCBs from a bare metal surface are presented in Table 2. On average, the AMSTAR solution was able to pull almost 70% of the PCBs into solution. This was an expected outcome because the glycol and surfactant components of the AMSTAR Dechlorination Solution can suspend the PCBs in a non-polar region of the otherwise polar, water based solution. The consistency in results of the wet AMSTAR samples (not field comparable as the samples were left in an AMSTAR solution that never dried) versus the dried AMSTAR application can most likely be attributed to an incomplete reconstitution of AMSTAR when the strips were soaked in water. Any small residue left behind most likely contained a small amount of PCBs which varied between samples. Also, the volume of water used to reconstitute the AMSTAR (20 mL) was much larger than that in the original aliquot (1 mL) which probably made it more difficult for the surfactant to optimally suspend the PCBs in solution.

The concentration of PCBs in the AMSTAR dried residue added to water plus the PCBs remaining on the metal coupons indicate a total recovery of PCBs approximately equal to that of the control samples. This means all of the PCBs removed from the metal strips by the AMSTAR solution were recovered and were not degraded.

It should be noted that a number of EPA procedures pertaining to the extraction of PCBs from solids require sonication of the samples for best extraction results. It is very likely the reason for the low total recovery of the control and subsequent AMSTAR treated samples is the lack of sonication to transfer the PCBs from the metal surface into hexane during the extraction step. However, since the controls were carried through the entire procedure, their extraction efficiency is the bench mark used to evaluate AMSTAR's treatment capabilities.

**Table 2: PCBs on Bare Metal Strips**

	PCBs Left on Metal Strip	PCBs in AMSTAR (residual reconstituted in water)	Total Recovery (Strip + AMSTAR Precipitate)	Avg Total Recovery	Std Dev
Control	28.05	1.49	29.54	30.28	0.74
	28.40	2.62	31.02		
	30.28	0	30.28		
Dried AMSTAR on Metal Test Strips	7.20	19.65	26.85	28.63	4.01
	8.79	*	*		
	12.73	18.16	30.89		
	6.99	*	*		
	27.76	5.11	32.87		
	9.22	14.71	23.93		
AMSTAR applied to strip in a closed vial (wet system)	2.44	26.06	28.50	35.14	9.11
	2.43	43.10	45.53		
	2.48	28.91	31.39		

\*Samples lost during recovery. Incomplete data not included in statistics.

\*\*All concentration are in mg/L.



## Painted Metal Coupons

While the bare metal strips treated with AMSTAR showed the ability of the product to remove PCBs from a non-complex surface, the same results were not observed when the PCBs were placed on a wet painted surface and allowed to dry in paint (Table 3).

**Table 3: PCBs in paint**

	Concentration Left in Paint			Average	Std Dev
Control	47.54	45.59	43.62	45.58	1.96
Hexane Extracted	46.28	43.73	44.33	42.71	3.32
	41.09	43.99	36.83		
Ethanol/Hexane Extracted	44.41	40.37	41.63	42.14	2.07
*All concentrations are in mg/L.					

Even though the PCBs were deposited on the top surface of a drying metal painted coupon, the AMSTAR product was not able to remove the PCBs from the highly non-polar, organic binder found in paint. The results indicate that the PCBs applied to a drying primer were not dechlorinated or degraded in any way, as better than 95% of the PCBs were recovered when the paint was extracted.

## Conclusions

The main objective of this study was to determine the ability of AMSTAR to remove and degrade PCBs from structural material. A series of tests was performed using Aroclor 1254 to test whether AMSTAR Dechlorination Solution was capable of degrading PCBs and if so, what byproducts did the dechlorination step leave behind. An additional set of experiments was run to determine the capability of AMSTAR to simply extract and not destroy PCBs from bare metal surfaces and painted surfaces. All analytical tests were performed in triplicate, with controls being carried through the entire laboratory testing procedure.

Our results indicate the AMSTAR product does not promote the dechlorination of Aroclor 1254, which is comprised of a number of PCB congeners. No degradation byproducts were detected by the GC-MS. AMSTAR does have the ability to extract PCBs from bare metal surfaces; however no degradation occurs once the PCBs enter the non-polar surfactant/solvent AMSTAR product. The residual, white dried AMSTAR product that remains after AMSTAR is applied to a surface still contains the non-degraded, Aroclor 1254 that was used in this laboratory evaluation. Based on the results of the painted metal coupon, the AMSTAR product was not able to remove the PCBs from the highly non-polar, organic binder found in paint. This indicates that the AMSTAR product's utility may be limited to removing surface deposits of PCBs as it cannot overcome the preference of PCBs for the coating's non-polar binder.

Another alternative for PCB extraction only work is to use d-Limonene or ethanol wipes on bare metal surfaces to remove and not degrade PCBs. The wipes can then be hauled off as TSCA level waste as needed. A quick evaluation of PCB-dosed metal coupons that were gently wiped with d-Limonene- or ethanol-soaked paper filters indicate over 40% PCB removal with a single application that was allowed to contact the metal coupon for only 10 seconds. This solvent wipe method is similar to the procedure outlined in 40 CFR 761 -Parts 125 & 130, which is used to evaluate the presence of PCBs on surfaces, and could be a more cost-effective PCB removal method (no degradation) for the DoD to consider over AMSTAR.